CHARGE NUMBER: 6908

PROGRAM TITLE: SMOKE CONDENSATE STUDIES

PERIOD COVERED: November 1-30, 1979

PROJECT LEADER: R. N. Ferguson

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A. CONDENSATE COLLECTION[1,2]

Experiments designed to trap the volatiles lost from impaction trapped (IT) and Elmenhorst cold-trapped (ECT) smoke during acetone processing were performed. The weights of volatiles collected in both experiments were in good agreement with that predicted by weights of trap contents made before and after usual processing. GC/MS profiling indicated the presence of the following compounds.

ECT volatiles: acetone, isomeric C_4-C_5 ketones and diones, cyclopentenone, furfural and homolog, furylmethylketone; methanol, furan homologs, cresol; acetonitrile and propionitrile; aliphatic dienes, limonene, dimethylbutene, methanoindene (alkylated) and norbornene; benzene and homologs, styrene and homologs, indene; pyridine and homologs, pyrazine homologs, hydroxypyridine;

IT volatiles (aqueous fraction): Methanol and allyl alcohol, acetone, isomeric C_4-C_5 ketones and diones, furfural, cyclopentenone; acetonitrile, propionitrile, cyanobutadiene; pyridine and homologs; methylpyrazine.

B. CHROMATOGRAPHY[3,4]

Usage of helium rather than nitrogen as carrier gas for the capillary gc system has eliminated the noise problem and considerably reduced the retention time of less volatile compounds, without degrading resolution. Chromatograms of a standard PAH mixture indicated preferential enhancement of earlier eluting compounds; this discrimination was attributed to problems with the injector. The specific cause for this effect is still under investigation. The RDANAL programs are being modified as necessary to handle capillary gc, with assistance from D. Jones.

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Development of an hplc method to analyze for harman and norharmane was initiated. Attempts to reproduce the literature method employing an isocratic solvent system were unsuccessful. Various gradient conditions were explored, but much more assay development is required.

In a collaborative study with W. Ryan, we have supplied a set of Grimmer fractions and base fractions prepared from 2R1 WSC.

C. CARBONYLS IN WSC[5]

Several carbonyl 2,4-DNPHs were quantitated using a reverse phase HPLC system comprised of an ODS-2 column, a CH_3OH-CH_3CN-H_2O gradient and 365 nm UV detection. Gas phase samples resulted from smoking 120 X6D8EUB cigarettes through a TPM pad into cold 1:1 (v:v) DMSO:H_2O. The TPM was dissolved in the same solvent. Derivatives were prepared and then these 2,4-DNPHs were isolated by the Seasorb-43 method. Compounds identified and the estimated amount of free carbonyl present (µg/cigt.) include

	TPM	Gas phase
Formaldehyde-DNPH	4.5	8.8
Acetaldehyde-DNPH	10.1	123.
Diacetyl-DNPH	0.6	2.0
Crotonaldehyde -DNPH	1.6	17.9
Furfural-DNPH	3.5	7.5
Unknown	10.2	17.2

Dose-response curves from *E. coli* assays indicate that gas phase is approximately twice as active as TPM.

D. MW 288[6]

Additional cuticular wax (7.86 g) has been obtained from green tobacco leaves (Coker 298) by dipping in chloroform. Scanning electron micrographs (100 x magnification) showed modifications to both leaf surfaces (upper and lower) and trichomes.

E. NITROSAMINES[7]

Additional samples have been analyzed for volatile nitrosamines for Project 6900. Extraction of solids at room temperature *versus* 70°C showed no differences so this modification of the procedure is now being used. Currently, efforts

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are centered on eliminating the background when commercially available Preptubes™ are used for aqueous samples. The use of these tubes will simplify analyses by eliminating the need for continuous extraction.

F. BASE FRACTIONS AND THE *SALMONELLA* ASSAY[4,8]

A gradient pH extraction of an X6D3IM base fraction was performed. The bases were dissolved in an 0.1 M citric acid solution and extracted with ether at 0.5 pH unit increments made by adding NH_4OH. The bulk of the mass was contained in the low pH (2.2, 2.7, and 3.2) and high pH (6.7, 7.7, 9.0) fractions. The twelve extracts, the base fraction, and the parent WSC were submitted for *Salmonella*/microsome assay.

The preparation of base and acids + neutrals fractions for fourteen IT WSCs was completed. The data from *Salmonella* assay of the samples and nicotine analysis of base fractions is now being interpreted.

G. BRIGHT TOBACCO FRACTIONATION[9]

As mentioned in the October report, amino acids, after acid hydrolysis, accounted for only about 25% of the nitrogen in the 70% ethanol extract of previously extracted bright tobacco. This is in agreement with other data that the protein extractants 5% KCl and 70% ethanol remove considerable nitrogenous material that is not simple protein.

H. REFERENCES

1. Hellams, R. Notebook No. 7335, pp. 139-140.
2. Kinser, R. Notebook No. 7153, p. 199.
3. Levins, R. Notebook No. 7351, p. 91.
4. Tafur, S. Notebook No. 7423, pp. 53-54.
5. Warfield, A. Notebook No. 7434, p. 23.
6. Katz, T. Notebook No. 7376, p. 146.
7. Baker, G. Notebook No. 7177, p. 191.
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